

CLAIMS

WHAT IS CLAIMED IS:

Claim 1 (currently amended): A process with operating conditions for preparing 1,1,1,3-tetrachloropropane, where the production efficiency of the desired product is improved comprising the steps of,

- a) mixing and heating carbon tetrachloride with ethene in a reactor in the presence of metallic iron, dissolved iron species, and trialkylphosphate co-catalyst to produce a continuous flow of reactor effluent containing a 1,1,1,3-tetrachloropropane product, and where the reactor temperature is between 80°C and 140°C, and the reactor pressure is between 10 and 300 pounds per square inch and the molar feed ratio, including recycle streams, of carbon tetrachloride and ethene is between 1.0 and 3.0.
- b) treating the reactor effluent from step a) in a sedimentation tube that separates coarse solid iron particles from the liquid and returns the coarse solid iron particles to the reactor, to produce by this separation a reactor effluent containing a decreased concentration of coarse solid iron particles,
- c) distilling the reactor effluent from step b) in a ~~refluxed evaporator~~ catalyst recovery unit to produce a continuous flow of overhead product containing an increased concentration of 1,1,1,3-tetrachloropropane and a continuous

flow of bottom product containing increased concentrations of dissolved iron and phosphorus-containing species that are active in the catalysis of the reaction to make 1,1,1,3-tetrachloropropane,

- d) recycling a portion of the bottom product from step c) to the reactor while purging the rest from the system,
- e) the overhead fraction of the refluxed evaporator being distilled to recover unconverted carbon tetrachloride and unconverted ethene,
- f) a portion of carbon tetrachloride and ethene being recycled back to the reactor, and
- g) the overhead fraction of the refluxed evaporator being distilled to recover purified 1,1,1,3-tetrachloropropane.

Claims 2- 23 (canceled).

Claim 24 (currently amended): The process in ~~claim 23~~ claim 1, where reactor temperature is between 90°C and 125°C.

Claim 25 (canceled).

Claim 26 (previously presented): The process of claim 25, where reactor pressure is between 40 and 200 pounds per square inch.

Claim 27 (canceled).

Claim 28 (previously presented): The process in claim 27, where the molar feed ratio, including recycle streams, of carbon tetrachloride and ethene is between 1.1 and 2.0.

Claim 29 (previously presented): The process in claim 1, where the molar feed ratio of tributylphosphate and metallic iron is between 1.0 and 2.0.

Claim 30 (previously presented): The process in claim 29, where the molar feed ratio of tributylphosphate and metallic iron is between 1.01 and 1.3.

Claim 31 (previously presented): The process in claim 1, where the molar feed ratio of dissolved iron and ethene is between 0.01 and 0.10.

Claim 32 (currently amended): The process in claim 31, where the molar feed ratio of dissolved iron and ethene is between ~~0.002 and 0.008~~ 0.02 and 0.08.

Claim 33 (previously presented): The process in claim 1, where the bottom temperature of the refluxed evaporator is between 70°C and 115°C.

Claim 34 (previously presented): The process in claim 33, where the bottom temperature of the refluxed evaporator is between most preferably between 80°C and 115°C.

Claim 35 (previously presented): The process in claim 1, where the refluxed evaporator pressure is between 40 TORR and 225 TORR.

Claim 36 (previously presented): The process of claim 1, wherein the trialkylphosphate co-catalyst is tributylphosphate or tripropylphosphate or triisobutylphosphate.

Claim 37 (previously presented): The process in claim 1, where the bottom fraction from the refluxed evaporator is fed into a second refluxed evaporator

- a. a substantial portion of the 1,1,1,3-tetrachloropropane remaining in bottom stream from the first refluxed evaporator is recovered in the overhead stream of the second refluxed evaporator and
- b. a portion of the bottoms from the second refluxed evaporator are recycled to the reactor.